Materials Scale-up and Cell Performance Analysis

Vince Battaglia
LBNL
DOE AMR
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Project ID #
ES029

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Overview

Timeline
- Start: June 2009
- End: September 2012
- Percent complete: 30%

Barriers
- Barriers addressed
  - High Cost (<150 $/kWh)
  - Low Energy Density (>230 Wh/l)

Budget
- Total project funding: $580 k
- Funding received in FY09: $290 k
- Funding for FY10: $290 k

Supports 1/8 Research Associates

Partners
- BATT PIs
  - Dillon (NREL)
  - Kumta (Pitt. U.)
  - Doeff (LBNL)
  - Lucht (U.R.I.)
  - Dudney (ORNL)
  - Thackeray (ANL)
  - Ceder (MIT)
  - Zaghib (HQ)
Objective

Evaluate New Materials Being Developed in the BATT Program Against DOE Goals and Baseline Performance Markers

- For FY '10 we expected to evaluate at least four new materials.

- Such evaluation provides guidance to the researchers as to what degree they have surpassed the baseline performance and how much farther we need to go toward meeting the DOE/USABC Performance Targets.

- Tracking progress is critically important to making progress.
1. Make inquiry to BATT PIs for new materials they deem ready for the next step
   - Scale-up to 10 g batch
   - Evaluation in full cell
2. Test in half cell against Li using electrode fabrication techniques developed in BATT program.
   1. If capacity density and first cycle irreversible capacity improve on baseline then go to next step.
   2. Measure rate capability at different C-rates at a reasonable loading. If improvements over baseline, then go to next step.
   3. Work with BATT cell modelers to design electrodes for full cells.
3. Evaluate cycle life in full cells.
   - Attempt to identify performance attributes and limitations.
4. Report performance results
   1. To PI
   2. At semi-annual ABRT meetings.
   3. If performance is favorable, at DOE AMR meeting.
Received 8 responses from BATT PIs expressing interest in scale-up and evaluation

– Completed evaluation of 4 materials.
– In the process of testing 3 materials
– With work with the last PI in the coming months.
# Technical Accomplishments

<table>
<thead>
<tr>
<th>Investigator</th>
<th>Institution</th>
<th>Material</th>
<th>Barrier</th>
<th>Feedback</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>G. Ceder</td>
<td>Massachussetts Inst. of Technology</td>
<td>High-rate LiFePO$_4$</td>
<td>High system cost</td>
<td>We made the material w/ their guidance</td>
<td>Cycle-life testing</td>
</tr>
<tr>
<td>M. Thackeray</td>
<td>Argonne National Laboratory</td>
<td>High-capacity NCM material</td>
<td>Low energy density</td>
<td>Sent us materials and electrode formulations</td>
<td>Cycle-life testing</td>
</tr>
<tr>
<td>N. Dudney</td>
<td>Oakridge National Laboratory</td>
<td>LiFePO$_4$ in carbon mat – no Cu cur. col.</td>
<td>High system cost</td>
<td>Sent us anodes</td>
<td>Tests complete (low cap. dens.)</td>
</tr>
<tr>
<td>M. Doeff</td>
<td>Lawrence Berkeley Nat. Laboratory</td>
<td>Al-doped NCM material</td>
<td>High material cost</td>
<td>We will make material w/ their guidance</td>
<td>To be initiated</td>
</tr>
<tr>
<td>P. Kumta</td>
<td>University of Pittsburgh</td>
<td>Si-C nanocomposite</td>
<td>Low energy density</td>
<td>Sent 1st gen anodes</td>
<td>Tests complete (high 1st cycle ICL)</td>
</tr>
<tr>
<td>K. Zaghib</td>
<td>Hydro-Québec</td>
<td>Lower cost LiFePO$_4$</td>
<td>High cost</td>
<td>Sent 50 g of powder and laminates</td>
<td>Cycle-life testing</td>
</tr>
<tr>
<td>A. Dillon</td>
<td>National Renewable Energy Lab.</td>
<td>High capacity MoO$_3$ anode</td>
<td>Higher energy density</td>
<td>Sent us anodes</td>
<td>Tests complete (high 1st cycle ICL)</td>
</tr>
<tr>
<td>B. Lucht</td>
<td>University of Rhode Island</td>
<td>LiPF$_4$C$_2$O$_4$ thermally stable salt</td>
<td>Poor high temperature performance</td>
<td>Sent us 10 g of salt</td>
<td>Tests complete (high 1st cycle ICL)</td>
</tr>
</tbody>
</table>
A comparison of two HQ materials: one received a year ago and one received 6 months ago. The new material was made via a less costly process.

When making comparisons, one needs to fabricate electrodes of identical loadings.
Technical Accomplishments

MIT Nano-LiFePO$_4$ Material

- We sent two scientists and Gao Liu to MIT for one week at the end of November during the MRS meeting in Boston
  - We made a batch of material there.
  - We were given ca. 1 gram of powder.
  - We were given an electrode laminate and Swagelok hardware for cell testing.
- Since then we have:
  - Made several 7 g batches of the material.
  - Characterized the materials with SEM, BET, and PSA.
  - Made laminates following their recipe using PTFE
  - Made laminates following our recipe using PVdF.
  - Exchanged testing results.
  - Prof. Ceder visited our lab in March.
  - Sent samples out for XPS analysis.
  - Been working to make higher rate electrodes.
  - Been working with the Molecular Foundry to do further surface analysis.

This has been a very open collaboration between all scientists involved!
Technical Accomplishments

SEM - Primary Particles

SEM at 100 nm
- For the HQ material, primary particles range from 50 to 500 nm
- For the MIT and LBNL, material primary particles are around ca. 25 nm
Technical Accomplishments

SEM - Electrodes

Electrodes

- HQ shows uniform mixing of additives and primary particles.
- For the MIT and LBNL, a fraction of the powder was still clumped together.
The MIT nano-LiFePO$_4$ is jet black.

- EDX indicated that there is some carbon on the surface.

- We sent this material out for XPS analysis, along with a sample of HQ’s material.
Technical Accomplishments

XPS & Ion Sputtering of MIT LiFePO$_4$

• Points of interest
  – Slight carbon coating of 1 to 2 nm, believed to be residue of FeC$_2$O$_4$·2H$_2$O precursor.
  – Oxygen and phosphorous are uniform and of the same ratio from surface to interior P:O = 1:4.
  – Accumulation of data suggests this sample is coated with 2 to 5 nm of Li$_3$PO$_4$. 
Technical Accomplishments

XPS & Ion Sputtering of HQ LiFePO₄

- **Points of interest**
  - Carbon coating 5 nm thick, believed to be an elemental carbon.
  - Oxygen and phosphorous are uniform and of the same ratio from surface to interior P:O = 1:4.
  - There may be a thin coating of Li₃PO₄ on the surface of this material as well, underneath the carbon.
## Collaborations

<table>
<thead>
<tr>
<th>Investigator</th>
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<th>Interaction</th>
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<tr>
<td>G. Ceder</td>
<td>MIT</td>
<td>We make material w/ their guidance</td>
<td>P. Kumta</td>
<td>U.Pitt.</td>
<td>Will send 30 to 100 g of 1st gen anodes</td>
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<td>ANL</td>
<td>Will send us materials and electrode formulations</td>
<td>K. Zaghib</td>
<td>H.Q.</td>
<td>Will send 30 to 100 g of powder and laminates.</td>
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<td>NREL</td>
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<td>U.R.I</td>
<td>Will send 10 g of salt</td>
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We are also working with V. Srinivasan of the Modeling Group of the BATT program to design cells.

P. Ross helped us with acquiring and interpreting the XPS data.

## Acknowledgment

The majority of this work was carried out by Jin Chong and Honghe Zheng.
Proposed Future Work

• Rest of this year
  – **MIT** material looks promising as a high rate material
    • Finish electrode processing and testing.
    • Work with BATT Modeling Group and MIT to determine best automotive application.
    • Design electrodes and test cells to that application.
    • We have additional experiments planned to understand why this sample is black.
  – **ANL’s** materials are in early phases of evaluation
    • Finish initial tests.
    • **Decision point** - If improvement over baseline materials, we will:
      – Work with BATT Modeling Group and ANL to determine best automotive application.
      – Design electrodes and test cells to that application.
  – **H.Q. laminates** are in the early stages of evaluation
    • Finish initial tests.
    • **Decision point** - If improvement over baseline materials, we will:
      – Work with BATT Modeling Group and ANL to determine best automotive application.
      – Design electrodes and test cells to that application.
  – Low-Co NCM is important, scale-up **LBNL’s** Al-substituted NCM.

• All milestones completed
Summary

- Eight BATT PIs answered the call for materials evaluation.
  - 4 sent laminates
  - 1 sent powders
  - 1 sent both
  - 2 asked us for assistance in scale-up

- Benefits and limitations of materials have been confirmed and conveyed back to PIs.

- New, low-cost HQ material performs as well as previous material.

- MIT material needs further processing to make good electrodes – this work has begun.

- Both MIT and HQ materials have coatings and perform well.
  - HQ’s has 100 nm primary particles with a 5 nm carbon coating that improves the electronic conductivity and leads to good electrode performance.
  - MIT’s has 30 nm primary particles with a 5 nm phosphate coating that mitigates secondary particle formation and minimizes solid-state diffusion limitations that leads to good electrode performance.